

US-PAT-NO: 5334310

DOCUMENT-IDENTIFIER: US 5334310 A

TITLE: Column with macroporous polymer media

DATE-ISSUED: August 2, 1994

US-CL-CURRENT: 210/198.2; 210/502.1 ; 210/635 ; 210/656 ;
95/88 ; 96/101

APPL-NO: 07/ 965181

DATE FILED: October 23, 1992

PARENT-CASE:

CROSS TO RELATED APPLICATION This application is a
continuation-in-part of U.S.
Ser. No. 07/779,929, filed Oct. 21, 1991, now abandoned.

----- KWIC -----

US PATENT NO. - PN:

5334310

Detailed Description Text - DETX:

After polymerization is complete, the solid macroporous
polymer plug is washed
to remove any porogenic solvent and with a suitable solvent
to dissolve any
soluble polymer present. Suitable washing solvents include
methanol, ethanol,
benzene, toluene, acetone, tetrahydrofuran, and dioxan.
This washing process
may be done in stages; for example, by washing with a
solvent, then with water
and then a solvent again, or by continuous washing with a
solvent. The washing
step is preferably carried out by pumping the solvent
through the tube filled

with the macroporous polymer.

Detailed Description Text - DETX:

Still another alternative relating to the washing step involves removing the polymerized plug from the tube, washing it with a suitable solvent, and then returning it to the tube. The returned polymer may be left as is or it may be swelled to occupy more space in the tube by washing the polymer with a swelling solvent such as tetrahydrofuran, 1,4-dioxan, toluene, or halogenated hydrocarbons.

Detailed Description Text - DETX:

One end of a stainless steel tube (4.6 mm inner diameter, 50 mm length) provided on both ends with fittings for attachment to a liquid chromatograph was closed with a steel nut stopper, the tube was purged with nitrogen and its other end was closed with a silicon rubber septum. A polymerization mixture was prepared by mixing 4.8 g glycidyl methacrylate, 3.2 g ethylene dimethacrylate, 10.8 g cyclohexanol, 1.2 g dodecanol, and 0.08 g azobisisobutyronitrile. The mixture was bubbled with nitrogen for 20 minutes to remove any oxygen present. 0.1 ml of the mixture was injected through the septum into the tube, and polymerization was started by heating in an thermostated oil bath at 70.degree. C. After 7 hours the tube was removed from the bath, cooled freely to room temperature, and the septum was removed. At this point the column contained a solid macroporous polymer plug 5 mm in length. After washing the polymer with methanol, both ends of the tube were closed with chromatographic nuts and the column attached to a HPLC

chromatograph. First, methanol was pumped through the column at different flow rates. The back pressure was 0.4 MPa at a flow rate 0.5 ml/min, 0.8 MPa at 1 ml/min and 1.6 MPa at 2 ml/min. The solvent was then changed to tetrahydrofuran and the back pressure was 0.2 MPa at 0.2 ml/min and 19 MPa at 2 ml/min. The dependence of back pressure on flow rate was found to be linear.

Detailed Description Text - DETX:

Into the same tube as used in Example I was injected 0.3 ml of a stock mixture consisting of 3 ml styrene, 1.2 ml divinylbenzene (85% of DVB), 6 ml benzylalcohol, and 0.05 g benzoylperoxide. After degassing, the tube was closed and polymerization proceeded at 70.degree. C. for 24 hours. Using a steel plunger an 18 mm long porous polymer plug was pushed out of the tube and washed for 4 days in several portions of methanol. After the washing the plug still fit in the original tube. When the solvent was changed to tetrahydrofuran, it swelled the plug. After about 20 minutes, the diameter of the plug exceeded the diameter of the tube. In order to shrink the plug to its original size, methanol was used for subsequent exchange of the solvent. The plug was then returned to the column and methanol was again exchanged back to tetrahydrofuran. The swelling in the column was monitored as a change of back pressure which increased from 0.3 MPa at the beginning when the rest of the tube was still filled with methanol to more than 10 MPa after exchanging some of the solvent. At that point the measurement was terminated to prevent any possible damage of the equipment.

Detailed Description Text - DETX:

The procedure of Example 1 is repeated except that the polymerization mixture is replaced by the formulation of Dutch patent application No. 6,803,739, example II, the tube is of flexible polytetrafluoroethylene, and the polymerization temperature is 60.degree. C. When the resulting tube and with its polymer plug is connected to a conventional chromatograph, essentially no flow of tetrahydrofuran through the plug is observed at pressures as high as 40 MPa (6,000 psi). When the plug is evaluated for pore size distribution as in Example 6 by cutting the tube apart to remove the plug, no pores larger than about 200 nm are found.